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IS 12875 (1989): Aluminium potassium sulphate (potash alum), photographic grade [CHD 5: Electroplating Chemicals and Photographic Materials]

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Indian Standard

ALUMINIUM POTASSIUM SULPHATE (POTASH ALUM), PHOTOGRAPHIC GRADE — SPECIFICATION

भारतीय मानक

एलुमिनियम पोटेशियम सल्फेट (पोटाश एलम), फोटोग्राफीय ग्रेड — विशिष्टि

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BUREAU OF INDIAN STANDARDS
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NEW DELHI 110002

July 1990 **Price Group 2**

Photographic Materials Sectional Committee, CDC 44

FOREWORD

This Indian Standard was adopted by the Bureau of Indian Standards on 29 December 1989, after the draft finalized by the Photographic Materials Sectional Committee had been approved by the Chemical Division Council.

This standard is one of a series of specifications for photographic grade chemicals which are commonly used in the processing of sensitized photographic materials. These specifications have been prepared to establish criteria of purity which will provide a practical and economical grade and prevent possible faulty processing which might be caused by chemicals of inferior quality and to furnish manufacturers, suppliers and processors with reliable and readily available specifications for photographic chemicals of satisfactory quality.

Aluminium potassium sulphate, also called potash alum, is used extensively in the processing of sensitized photographic materials. It has a chemical formula of $Al_2(SO_4)_3.K_2SO_4.24H_2O$ and molecular mass of 948. Potash alum used for dyeing, printing fabrics, paper, vegetable glue, purification of water, and for analytical laboratory use is covered under IS 258 : 1967 'Specification for potash alum (*first revision*)', which so far covered photographic grade also. With the printing of this standard, photographic grade will be subsequently deleted from IS 258 : 1967.

In formulation of this standard, assistance has been drawn from ISO 3620 : 1976 'Photographic grade aluminium potassium sulphate dodecahydrate — Specification.'

For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

Indian Standard

ALUMINIUM POTASSIUM SULPHATE (POTASH ALUM), PHOTOGRAPHIC GRADE — SPECIFICATION

1 SCOPE

1.1 This standard specifies the requirements and methods of sampling and test for aluminium potassium sulphate (potash alum) for use in the processing of sensitized photographic materials.

2 REFERENCES

2.1 The following Indian Standards are necessary adjuncts to this standard:

IS No.	*Title*
321 : 1964	Specification for absolute alcohol (*revised*)
1070 : 1977	Specification for water for general laboratory use (*second revision*)
4905 : 1968	Methods for random sampling

3 REQUIREMENTS

3.1 The material shall be in the form of a white powder or colourless crystals.

3.2 Appearance of Solution

A solution containing 5 g of the sample in 100 ml of distilled water shall be clear and colourless and free from sediment other than a slight flocculence.

3.3 The material shall also comply with the requirements given in Table 1 when tested by the methods given in Annex A. Reference to the relevant clauses of Annex A is given in col 4 of Table 1.

4 PACKING AND MARKING

4.1 Packing

For bulk packing, the material shall be packed in flexible containers such as of paper, plastic, fibreboard and jute with suitable liner. For smaller packing, the material shall be packed in moisture-proof glass/polyethylene bottles.

4.2 Marking

Each package shall be legibly and indelibly marked with the following information:

a) Name and grade of the material,
b) Net mass of the material,
c) Indication of the source of manufacture,
d) Batch number or lot number in code or otherwise, and
e) Month and year of manufacture.

5 SAMPLING

5.1 The method of preparation of test sample and the criteria of conformity shall be as prescribed in Annex B.

Table 1 Requirements for Aluminium Potassium Sulphate (Potash Alum), Photographic Grade

(*Clauses* 3.3 *and* B-2.2)

Sl No.	Characteristic	Requirement	Method of Test (Ref to Clause No. in Annex A)
(1)	(2)	(3)	(4)
i)	Assay, *Min*	99·5	A-2
ii)	Matter insoluble in water, percent by mass, *Max*	0·015	A-3
iii)	Heavy metals (as Pb), percent by mass, *Max*	0·005	A-4
iv)	Iron (as Fe), percent by mass, *Max*	0·01	A-5

ANNEX A

(*Clause* 3.3)

METHOD OF TEST FOR ALUMINIUM POTASSIUM SULPHATE (POTASH ALUM), PHOTOGRAPHIC GRADE

A-1 QUALITY OF REAGENTS

A-1.1 Unless specified otherwise, pure chemicals and distilled water (*see* IS 1070 : 1977) shall be employed in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

A-2 ASSAY

A-2.0 Two methods are prescribed, namely:

a) Gravimetric method, and

b) Titration with EDTA solution.

A-2.1 Gravimetric Method

A-2.1.0 *Principle*

Potash alum is estimated by precipitating aluminium with ammonium hydroxide and calculating the weight of potash alum from the ignited residue.

A-2.1.1 *Reagents*

A-2.1.1.1 *Ammonium chloride*, solid.

A-2.1.1.2 *Methyl red indicator*

Dissolve 0·30 g of water soluble methyl red in water and dilute it to one litre.

A-2.1.1.3 *Dilute ammonium hydroxide*, approximately 5 N.

A-2.1.1.4 *Ammonium nitrate solution*, approximately 2 percent (*m*/*v*).

A-2.1.2 *Procedure*

Weigh accurately about 2 g of the sample and dissolve in 300 ml of water. Filter to remove any undissolved residue and thoroughly wash the filter paper with hot water. Add to the filter and washings about 5 g of ammonium chloride. Boil the solution and add two drops of methyl red indicator. Add ammonium hydroxide solution, drop by drop, until the colour changes to distinct yellow. Boil for two minutes and filter immediately through Whatman filter paper No. 41 or its equivalent. Wash the precipitate of mixed oxides of iron and aluminium with hot ammonium nitrate solution till the filtrate is free from chlorides. Ignite the precipitate to a temperature of 1 100 to 1 200°C in a tared silica crucible to constant mass.

A-2.1.3 *Calculation*

$$\text{Potash alum, percent by mass} = \frac{m \times 9{\cdot}307 \times 100}{M}$$

where

m = mass of the ignited residue, and

M = mass of sample taken.

A-2.2 EDTA Method

A-2.2.1 *Reagents*

A-2.2.1.1 *Disodium ethylene diamine tetra-acetate (EDTA) solution*

0·1 M. Dissolve 37·225 g of disodium ethylene diamine tetra-acetate dihydrate in water and make the volume to one litre in a volumetric flask.

A-2.2.1.2 *Absolute alcohol, see* IS 321 : 1964.

A-2.2.1.3 *Dithizone indicator*

0·025 percent in absolute alcohol. It shall be prepared afresh.

A-2.2.1.4 *Acetic acid - ammonium acetate buffer solution*

Dissolve 7·709 g of ammonium acetate in 100 ml of water. Mix 6·005 g acetic acid in 100 ml of water. Mix both the solutions together. The final solution is in the concentration of one mole of ammonium acetate and one mole of acetic acid per litre.

A-2.2.1.5 *Standard zinc chloride solution*, 0·1 M.

A-2.2.2 *Procedure*

Dissolve 2 g of the sample in 25 ml of water, add 50 ml of EDTA solution followed by 10 ml of buffer solution, and then 85 ml of absolute alcohol and 3 ml of dithizone indicator. Titrate the excess of EDTA solution with standard zinc chloride solution until the colour changes from blue to reddish-purple.

A-2.2.3 *Calculation*

Calculate the potash alum content of the material on the assumption that one millilitre of 0·1 M EDTA solution is equivalent to 0·047 44 g of potash alum [as $Al_2(SO_4)_3.K_2SO_4.24H_2O$].

A-3 DETERMINATION OF MATTER INSOLUBLE IN WATER

A-3.1 Procedure

Weigh accurately about 25 g of the sample. Dissolve in 250 ml of water by warming, if necessary, and by stirring the solution well. Filter through a tared filter paper or Gooch crucible or a sintered glass crucible (G. No. 4). Wash the residue thoroughly with water till it is free from sulphates, and dry to constant mass at 110 ± 5°C.

A-3.2 Calculation

$$\text{Matter insoluble in water, percent by mass} = \frac{100\,m}{M}$$

where

m = mass in g of the residue obtained, and

M = mass in g of the sample taken for the test.

A-4 DETERMINATION OF HEAVY METALS (as Pb)

A-4.0 Principle

Heavy metals are determined by comparing the colour produced by the material with hydrogen sulphide against that produced by a standard lead solution.

A-4.1 Apparatus

A-4.1.1 *Nessler Cylinders*, 50-ml capacity.

A-4.2 Reagents

A-4.2.1 *Standard Lead Solution*

Dissolve 1·60 g of lead nitrate in water, add 1 ml of concentrated nitric acid and make up the volume to 1 000 ml. Transfer exactly 10 ml of the solution into a volumetric flask and dilute it again with water to 1 000-ml mark. One millilitre of this solution contains 0·01 mg of lead (as Pb). The dilute solution shall be prepared afresh.

A-4.2.2 *Acetic Acid*, approximately 1 *N*.

A-4.2.3 *Hydrogen Sulphide Solution*, saturated.

A-4.3 Procedure

Weigh exactly 1·0 g of the sample and add 15 ml of water. Add 0·5 ml of acetic acid and 10 ml of hydrogen sulphide solution. In the second Nessler cylinder, carry out a control test using 5 ml of standard lead solution and the same quantities of other reagents. Dilute the contents of each tube to 50 ml and shake well. Compare the colour produced in the two cylinders.

A-4.3.1 The limit prescribed in Table 1 shall be taken as not having been exceeded if the intensity of colour produced with the material is not greater than that produced in the control test.

A-5 DETERMINATION OF IRON (as Fe)

A-5.1 Apparatus

A-5.1.1 *Nessler Cylinders*, 50-ml capacity.

A-5.2 Reagents

A-5.2.1 *Acetate Buffer Solution, pH 5·0*

Dissolve 23 g of anhydrous sodium acetate in 58 ml of 2 M acetic acid and dilute to 1 000 ml with water. Adjust the final *p*H to 5·0 ± 0·1 with glacial acetic acid or 100 g/l sodium hydroxide solution.

A-5.2.2 *Standard Iron Solution*

Weigh 0·702 g of ferrous ammonium sulphate [$FeSO_4.(NH_4)_2SO_4.6H_2O$] and dissolve in 10 ml of dilute sulphuric acid. Dilute with water to make up the volume to 1 000 ml. Transfer 10 ml of this solution, and again dilute with water to make up the volume to 100 ml. One millilitre of this solution is equivalent to 0·01 mg of iron (as Fe).

A-5.2.3 *1, 10-Phenanthroline Reagent Solution*

Thoroughly mix equal volumes of aqueous solution of acetate buffer, hydroxyl ammonium chloride (10 percent *m*/*v*) and 1, 10-phenanthroline (1 g/l) in the specified order.

A-5.3 Procedure

Weigh accurately about 0·5 g of the sample. Transfer to one of the Nessler cylinders and dissolve in 25 ml of water. Also transfer 5 ml of the standard iron solution to the other Nessler cylinder, add 20 ml of water. Add 5 ml of 1, 10-phenanthroline reagent solution to each, mix and allow to stand for 10 minutes. Dilute each to 50 ml and mix well.

A-5.3.1 Compare, in the Nessler cylinder, the colour produced in the test and control solutions.

A-5.3.2 The sample shall be taken as conforming to the requirement when the colour produced in the test solution is not greater than that produced in the control solution.

ANNEX B

(*Clause* 5.1)

SAMPLING OF ALUMINIUM POTASSIUM SULPHATE (POTASH ALUM), PHOTOGRAPHIC GRADE

B-1 LOT

B-1.1 In a consignment all the containers of the same grade of material and produced by one manufacturer shall constitute a lot. Each lot shall be separately inspected for deciding the conformity to the requirements of the specification.

B-2 SCALE OF SAMPLING

B-2.1 The number of containers to be selected from a lot shall be in accordance with Table 2 and these shall be selected at random. For random selection methods guidance may be had from IS 4905 : 1968.

B-2.2 For description as well as appearance of solution each container of the sample shall be opened and examined for relevant requirements. For other characteristics, a composite sample shall be prepared by taking adequate quantity from each of the sample containers and thoroughly mixing them by suitable means. This composite sample shall be tested for the relevant requirements given in Table 1.

B-2.3 The lot shall be considered conforming to the requirements of this specification if both individual as well as composite samples pass the corresponding tests.

Table 2 Scale of Sampling

(*Clause* B-2.1)

Lot Size	Sample Size
Up to 25	2
26 to 50	3
51 to 100	4
101 and above	5

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